THE EFFECT OF EMERGING ADMIXTURES ON THE CORRELATIONS BETWEEN WORKABILITY TESTS ON MORTAR FOR 3D PRINTING

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Abstract:

Additive manufacturing for cement-based materials is gaining interest these past few years. Printing concrete has the potential to remove the time allowed to casting and molding. Nevertheless, new issues specific to 3D printing emerge. For example, the preservation of the mechanical properties and the stability of the printed layers. The objective of this paper is to identify the characteristics needed for a printed mortar to fulfill its role and propose a mixture that fills the required characteristics. The mixtures tested are composed of various combinations of superplasticizer (SP), accelerator (A), nanoclay (C), strength-enhancing admixture (X) and viscosity-modifying admixture (VMA). Three tests are conducted to study the impact of several admixtures on the capacity of the mortar to keep its shape. The first one is the measure of the slump with an Abrams cone test at a smaller scale. The second one is the flow test of the ASTM C1437. The third one is a stability test designed to simulate the load of printed layers. To propose a value of stability of the mortar, the deformation of a fresh cylinder of 35 mm height and 60 mm diameter is recorded under a force of 100 N progressively applied. The correlation between the tests are calculated. Results indicate that conventional tests are not effective with emerging admixtures like the strength-enhancing admixture. Whereas good correlation are made between slump of mortar and its capacity to maintain its shape under a load, the shape stability test.

1. INTRODUCTION

Being able to predict the comportment of a cementitious material in its fresh state is one of the main issues of 3D printing. While classic manufacturing of concrete structure requires formwork and seeks a concrete fluid enough to fill it, the 3D printing material has to be stiff to stay still during the process. Hence, in order to study the comportment of cementitious materials in their fresh state “slump test” or “flow test” are conducted. Those tests differ considering if the material is cement paste, mortar or concrete. The slump test for concrete is conducted with the Abrams cone [1], the procedure is described in the ASTM C143 [2]. The mini-slump test is used on cement paste and the mold can take various shapes depending on the study [3-6]. The more common is a smaller version of the Abrams cone for concrete [3, 7, 8], which keeps the same proportions, 3-2-1 respectively for the height, the bottom opening and the top opening. Most of the studies focus on the link between cement paste flow and concrete behavior [6], thanks to rheological measurement at large scale. However, rheometers are expensive devices and difficult to implement in situ. In this paper, we propose three tests, one for the flow, one for the slump and a last one is a specially designed test to get information about the behavior of the mortar in a 3D printing like situation. The results of each test are computed and regressions show that each test is linked to the other linearly. The best correlation is obtained between the stability test, which gives a deformation under a load, along with the slump test. Moreover the presence of different admixture is studied. Finally, being able to describe other results thanks to the Abrams cone test could be an asset to quickly describe a mortar without having to conduct long or difficult experiments.

2. MATERIALS

2.1 Materials properties

A binary cement with silica fumes (GUb-8SF) is used in this study. Its specific gravity is 2.8. The sand is a local sand with a specific gravity of 1.65. The water used is normal tap water.

2.2 Admixtures

Several admixtures are tested. The solid content of the admixtures is determined according to ASTM C494 [9]. Results are presented in Table 1. A superplasticizer (SP) is added to increase the workability of each mixture. The accelerator (A) also increases the workability. The strength-enhancing admixture (X) is a CSH-seed admixture; it is known to improve cement hydration and enhances workability. Nanoclays (C) is used to increase the stability of the mix. For each mix the procedure of the addition of the admixtures is always the same: they are added to the water in the mixer.

Table 1: Admixtures

Admixture	Residue by oven drying (%w/w)	Density
SP	25.8	1.050
A	47.2	1.350
X	30.1	1.120
VMA	1.04	1.002
C	N/A	1.000

2.3 Methodology

2.3.1 Small Abrams cone

A cone shaped as the Abrams cone used for concrete slump test is used and its dimensions are proportional to its bigger version. It is 150 mm high, the diameter of the bottom and the top opening are respectively 100 mm and 50 mm. The cone is filled with three layers of mortar 2 minutes after the end of the mixing procedure. Each layer is approximately one third of the volume of the mold and is tamped 25 times with a rod as recommended in the ASTM C143 for Slump of hydraulic-Cement Concrete [2]. The mortar is cut off to a plane surface flush with the top of the mold. The cone is removed slowly enough to avoid inertia issues (< 0.005 $m.s^{-1}$) [4, 5]. The test is conducted on an acrylic glass plate as proposed by Tan *et al*. [5] marked with a 2 x 2 cm^2 grid. The slump is measured between the maximum height of the mold and five points on the surface of the cone, as illustrated on Figure 1.

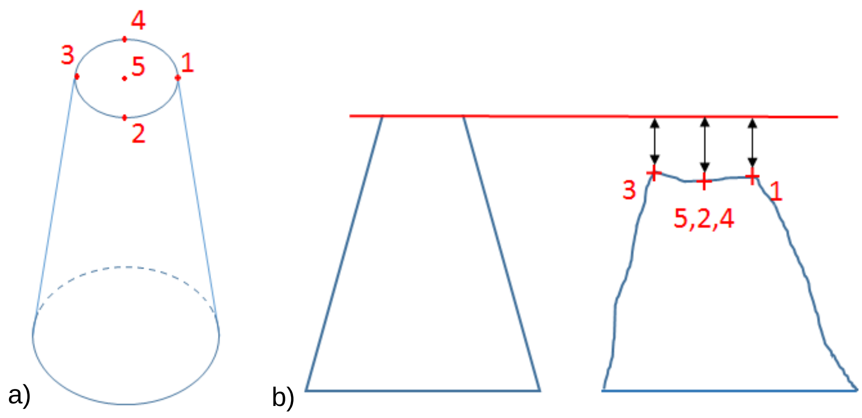

Figure 1: a) Perspective view of the mold b) Slump test and measurement

2.3.2 Flow test ASTM C1437

Another test is described by the ASTM C1437 [10] in order to get information about the consistency of hydraulic cement mortar. The mortar is unmolded on a special table, which is dropped 25 times in 15 s. The flow of each mix is recorded with the caliper specify in the standard along four diameter scribed on the table. This test was conducted 1’40’’ after the end of the mixing procedure.

2.3.3 Stability Test

The stability of the fresh mortar is determined using a method inspired by Kazemian *et al.* and Perrot *et al.* [11, 12] that proved that this kind of procedure could simulate the stacking of several layers on each other. A 35 mm high and 60 mm diameter cylinder is molded and immediately unmolded. A plastic tape placed on the wall of the mold still maintained the cylinder until the beginning of the test. After removing the tape, a thin galvanized steel plate is gently put on the top of the cylinder in order to allocate the forthcoming load on the surface. Then a photograph of the cylinder is taken and the height is computed with the picture processing software ImageJ, using a ruler placed on the photo to calibrate the scale (Figure 2.a). The test is conducted controlling a hydraulic press squeezing the cylinder at a constant rate of 1 mm/min. The force is recorded with a 0 to 100 N load cell at a sampling rate of 5 Hz. The press is stopped at 95 N and another photograph of the cylinder is taken. Its height is computed following the same procedure as for the first picture (Figure 2.b). This test is conducted 10 min after the end of the mixing procedure.

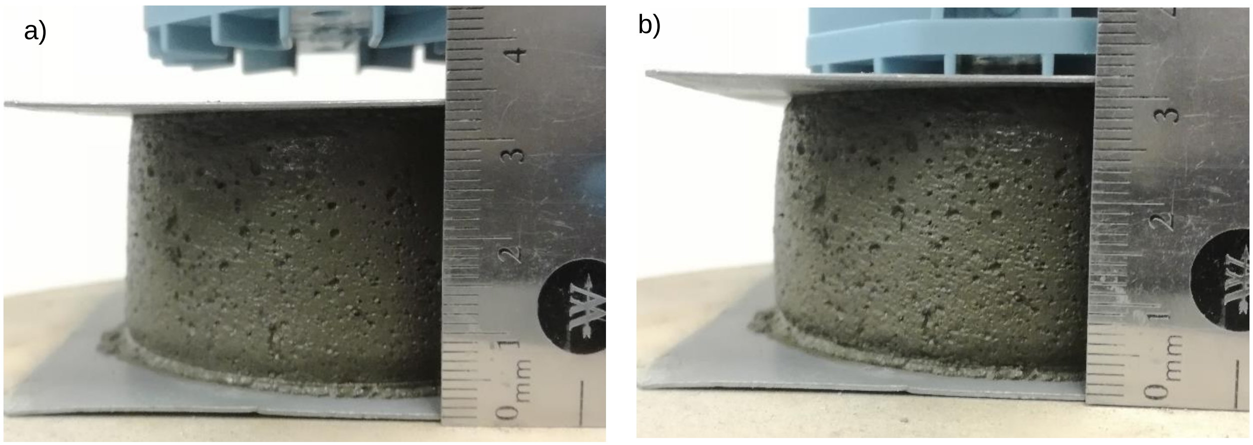

Figure 2: Cylinder of mortar a) before conducting the stability test, b) after conducting the stability test

2.3.4 Mix design of mortars

For the confection of each mix of mortar, the water / cement ratio is kept at 0.345 and superplasticizer (SP) is added at 0.26 % by weight of cement. For each mix the admixture residue by oven drying is determined and the corresponding amount of water present in it is subtracted to the total water added. Considering the fact that the final goal is to have a printable mortar, the sand / cement ratio is kept at 1.8 to optimize the amount of paste in the mix in order to enhance its pumpability [13-17]. A 2-level full-factorial design is created to allow each of the four admixtures to be tested in all configurations. This results in 2^4 different mixes. All the mixes are reported in Table 2.

Table 2: Mix design of mortars

Materials (kg/m3)				Admixture (%w/w)				
N° mix	Gub-8SF	Sand	Water	SP	X	A	C	VMA
M1	753	1355	254	0.26	-	-	-	-
M2	753	1355	251	0.26	-	-	-	0.004
M3	753	1355	254	0.26	-	-	0.5	-
M4	753	1355	251	0.26	-	-	0.5	0.004
M5	753	1355	248	0.26	-	0.7	-	-
M6	753	1355	245	0.26	-	0.7	-	0.004
M7	753	1355	248	0.26	-	0.7	0.5	-
M8	753	1355	245	0.26	-	0.7	0.5	0.004
M9	753	1355	249	0.26	0.3	-	-	-
M10	753	1355	246	0.26	0.3	-	-	0.004
M11	753	1355	249	0.26	0.3	-	0.5	-
M12	753	1355	246	0.26	0.3	-	0.5	0.004
M13	753	1355	243	0.26	0.3	0.7	-	-
M14	753	1355	240	0.26	0.3	0.7	-	0.004
M15	753	1355	243	0.26	0.3	0.7	0.5	-
M16	753	1355	240	0.26	0.3	0.7	0.5	0.004

2.3.5 Analysis of the results

The fact that a full-factorial design is used allows us to try to find correlation between the presence of some admixtures and the behavior of the mixes. Linear regressions are conducted to identify relationships between the admixtures and the results. The coefficient of determination and the regression equation determined. In addition, the confidence intervals of the regression line itself are computed.

3. RESULTS AND DISCUSSION

3.1 Abrams cone slump and ASTM C1437 flow

3.1.1 Raw results

Once the cone was removed mixes were quite immediately static. Hence, the slump could be measured 10 minutes after the cone removal. The results of flow and slump are gathered in Table 3.

Table 3: Abrams cone slump and ASTM C1437 flow results for mortar

Mix	Mix1	Mix2	Mix3	Mix4	Mix5	Mix6	Mix7	Mix8
Abrams cone	26	36.6	31.6	20.2	70	48	50	35.8
Standard deviation	2.61	2.06	1.02	2.71	3.79	1.9	4.38	5.15
ASTM C1437	101	96	97	85	115.5	110	107	97
Mix	Mix9	Mix10	Mix11	Mix12	Mix13	Mix14	Mix15	Mix16
Abrams cone	24.6	27.8	21.6	25	30.2	27.6	23.8	13
Standard deviation	4.03	2.79	2.73	2.37	4.58	2.87	4.12	0.32
ASTM C1437	95	92.5	92	84.5	95.5	92	95.3	75.5

3.1.2 Correlation between slump and flow

In order to highlight the link between the flow and the slump of the mortar, a linear regression has been computed between the flow and the slump of 16 mixes (Figure 3.a). The regression equation and coefficient of determination R^2 are recorded in Table 4. On the Figure 3.b the same data are plotted specific to each admixture. The regression equation and the coefficient of determination are recorded too in Table 4. First, we can observe that the mixes with the viscosity-modifying admixture (VMA) give a high correlation for those tests. Then mixes with the accelerator (A) and the nanoclay (C) still have a good correlation. This implies that those admixtures affect the flow and the slump of the mixes the same way. Therefore we can conclude that the presence of A, C or VMA allows to explain the flow of the mortar thanks to its slump linearly. In the opposite the CSH-seed admixture (X) leads to a low correlation, as a result the flow cannot be explained linearly by the measure of the slump for mix-containing X.

Equations can be drawn from these regressions. They are of the following form:

[1] $Flow = \alpha * Slump + \beta$

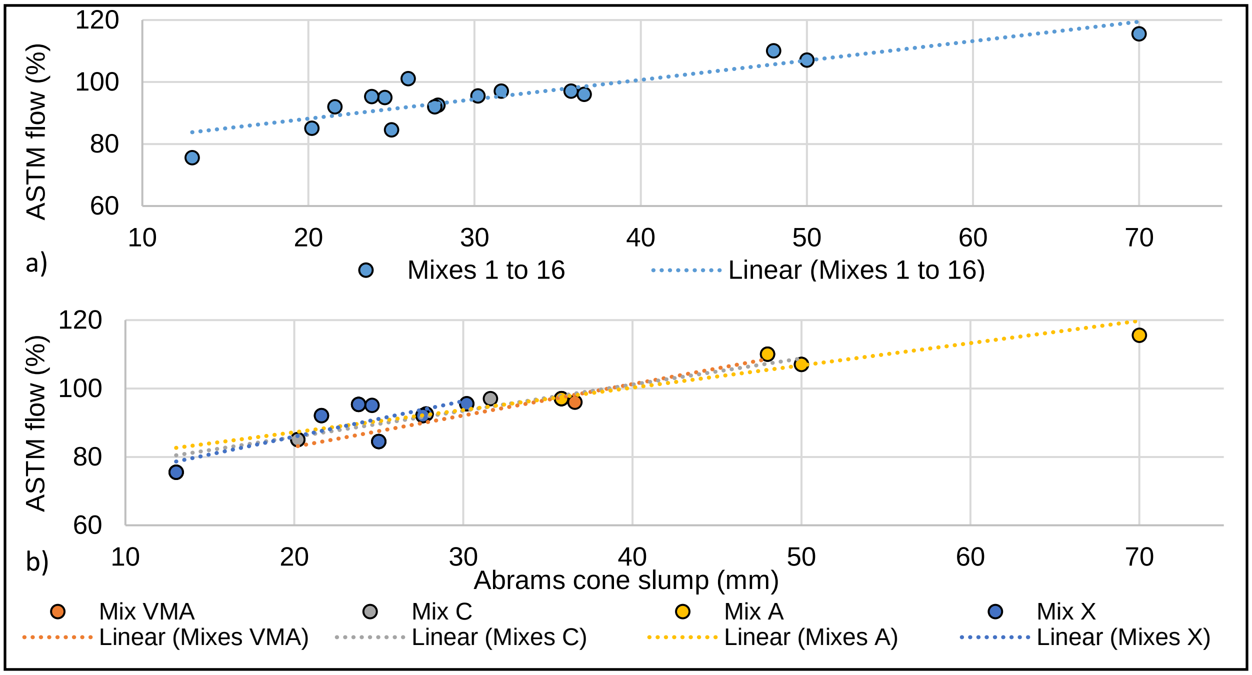

Figure 3: ASTM C1437 flow versus Abrams cone slump for a) Mixes 1 to 16 and b) Each admixture independently

Table 4: Equation and coefficient of determination for the different regressions between slump and flow

Mixes involved	Regression equation	R^2
Mixes 1 to 16	Flow = 0.6257*Slump + 75.661	0.79
VMA	Flow = 0.9159*Slump + 64.643	0.94
C	Flow = 0.7655*Slump + 70.515	0.81
A	Flow = 0.6516*Slump + 74.17	0.89
X	Flow = 1.0403*Slump + 65.113	0.62

When making regression it is important to be aware of the limitations of the results. To this end confidence intervals are determined. For the regression line, the value of the Flow is estimated for a known Slump at a level of confidence of 95%. Figure 4 shows the confidence intervals for the regression line when all mixes are involved. We can see that some points are out of the intervals. Those points are mainly mixes with X (Fig. 4).

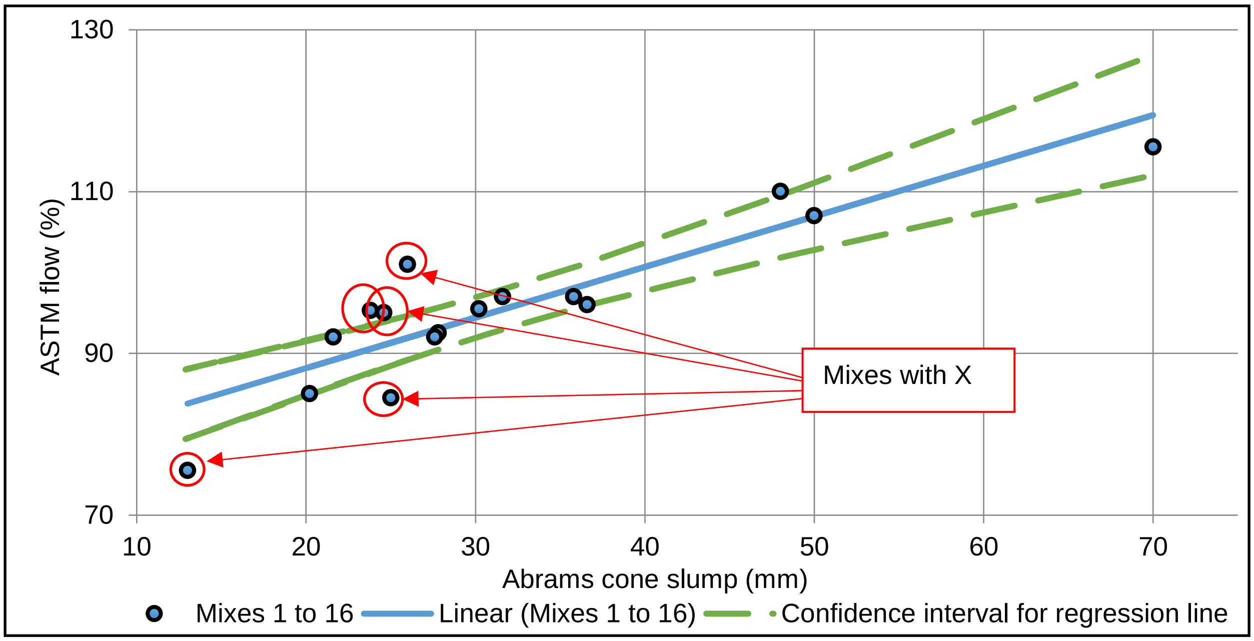

Figure 4: Confidence intervals when all mixes are involved for the regression line concerning flow versus slump.

3.2 Abrams cone slump and deformation with stability test

To investigate the link between slump and deformation a linear regression have been conducted. As the previous flow test the stability test implies greater shear stress in the material compare to the slump test. For the first one the table is dropped 25 times and for the second one a load is apply on the mortar. Those stresses on the mortar could be the explanation of the nonlinearity when testing the mixes containing X. Here the coefficient of determination for those mixes is only 0.33 while those for VMA, C and A, mixes are greater than 0.85 (Table 5). Once again, we can conclude that the CSH-seed admixture does not have the same impact on the mortar in function of the test.

Table 5: Equation and coefficient of determination for the different regressions between deformation and slump

Mixes involved	Regression equation	R^2
VMA	Slump = 4,3236*Deformation - 4,723	0,94
C	Slump = 4,5358*Deformation - 6,126	0,93
A	Slump = 4,6828*Deformation - 2,7722	0,86
X	Slump = 2,5504*Deformation + 8,1158	0,33
Mixes 1 to 16	Slump = 4,9598*Deformation - 7,6318	0,84

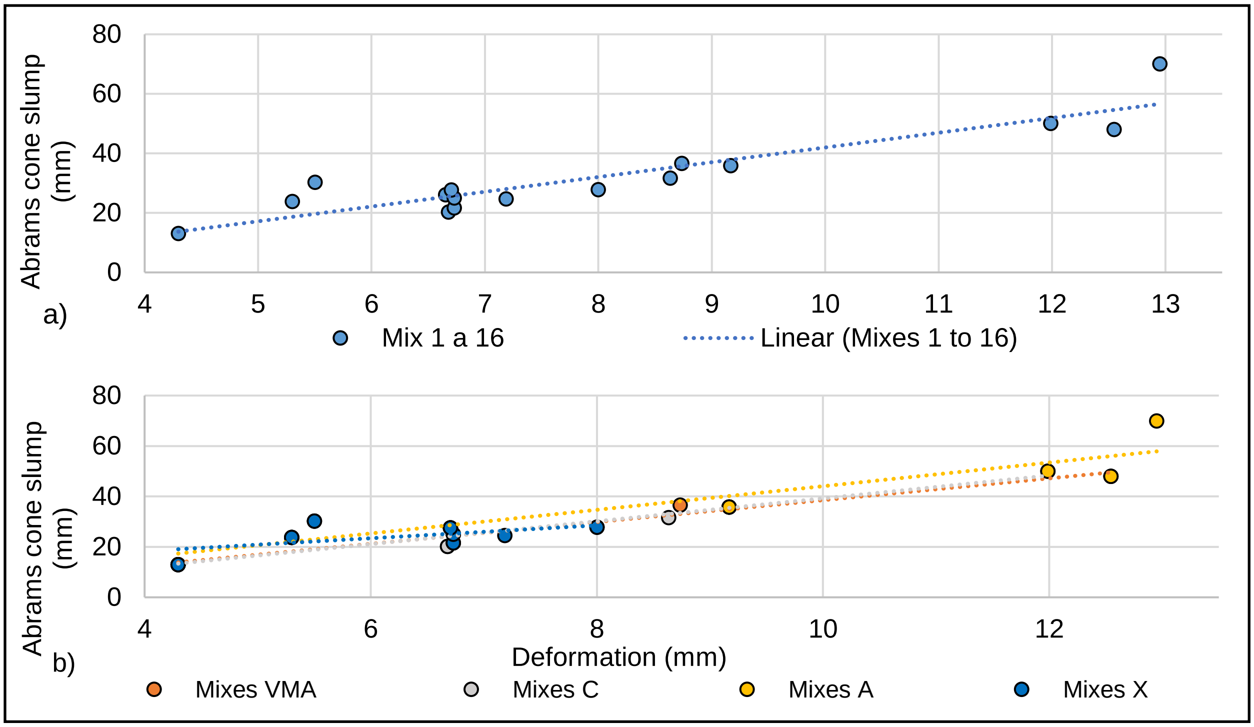

Figure 5: Abrams cone slump versus deformation with stability test for a) Mixes 1 to 16 and b) Each admixture independently

The computation of the coefficients of determination and the regression equations in Table 5 leads to the same conclusion about the CSH-seed admixture. The correlation between flow and deformation is the weaker for mix-containing X and the higher for mix-containing VMA. This is the same observation as for the previous results. Consequently, we can suppose that VMA admixture enhances linear correlation between those three tests while X admixture tends to make the mortar behavior unpredictable. Those results are supported by the study between flow and deformation not presented in this paper. A previous research about VMA in concrete proved that its presence increase the flow time and decrease the slump [18, 19]. This is relevant with the fact that when the slump is lower it is the same for the flow. This is explained by the action of fixing free water of the VMA admixture and its capacity to enhance cohesion.

For the confidence interval of the regression line (Fig. 6.b) we can observe that several points are out of the boundaries. Most of them are X mixes (Mixes 10, 11, 13 and 15). Slump and deformation are less related for those mixes. Once again, the presence of CSH-seed admixture seems to prevent from connecting results. The mixing procedure has to be modified in order to incorporate better this admixture.

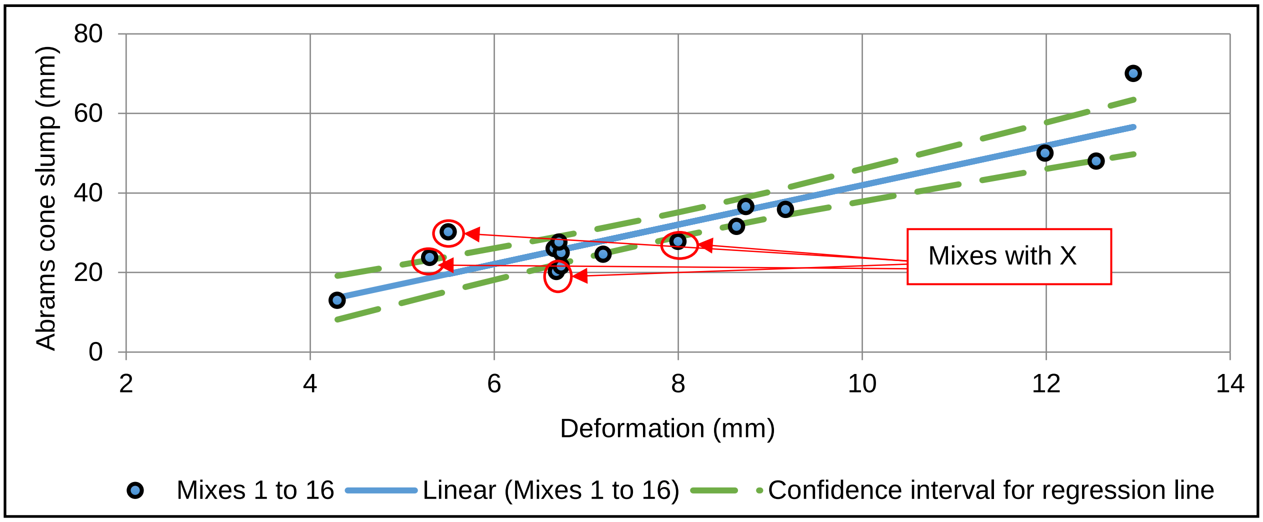

Figure 6: Confidence intervals when all mixes are involved for the regression line concerning slump versus deformation

3.3 Requirements of mortar mixtures for 3D printing

While 3D printing process needs the mixture to be balanced between its physical characteristics like flow or slump here we have separately study several measurements. It should be noted that encountering one or two of required values for printing cannot grant without fail the printability. Some values of flow of 119 % or 118 % lead respectively to a collapse of the printed structure or at least to a strong deformation while for a flow of 113 % or 116 % the deformation is considered acceptable [11]. However, a mortar with a very high fluidity can still be printed. For Zhang et al. the optimal mix was the one permitting to print approximately 22 layers or 260 mm without collapsing. This lead to a mixture having a flow of 168 % [20]. Equivalent values of deformation for our cylinder (Tab. 6) give us clues about the critical decrease. Hence, the value of 6.3 mm is taken to ensure stability. Moreover the equation linking the slump and the deformation leads to a second threshold value, which is 26.3 %. Mixes are here considered critical when above the threshold values, this allows to draw a map of the acceptable mixes (Fig. 7). The mixes circled in black are the mixes which could be kept to further study on their printability. Finally the flow of our mixtures is always lower than the critical flow of 116 % but a flow too low could lead to a mortar too stiff which is not desirable for printing [11, 17]. Therefore we could also exclude the 16th mix.

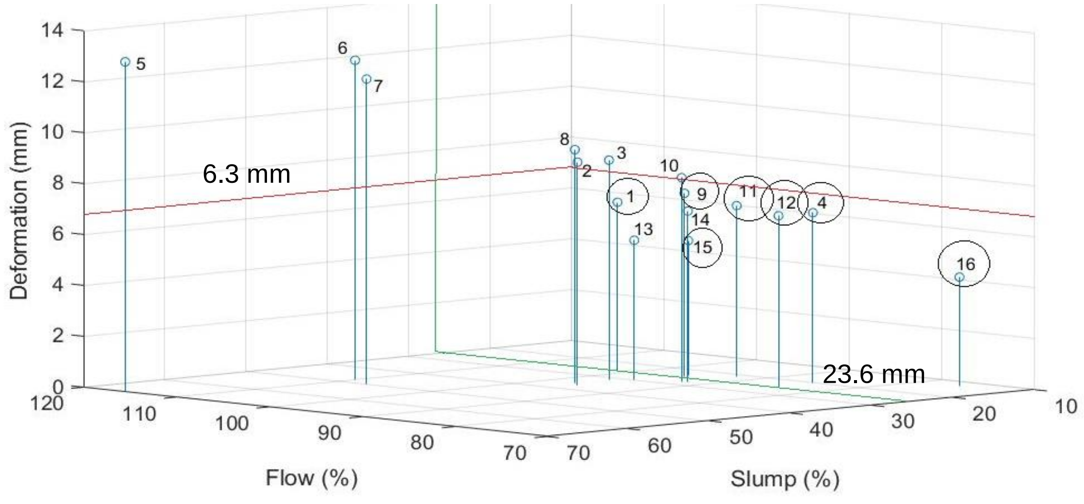

Figure 7: Mixes from 1 to 16 considering their values of flow, slump and deformation (acceptable mixes are circled)

Table 6: Values of flow and deformation from literature for printed mortars

	Kazemian 2017		Results		Zhang 2018	
Flow (%)	Diminution of height of a printed layer (%)	Deformation in height of the 80 mm high cylinder (%)	Equivalent for a 35 mm high cylinder (mm)	Slump from regression (mm)	Height of the printed structure (mm)	Decision
119	collapse	48	16.8	75.7	-	Rejected
116	6,7	18	6.3	23.6	-	Acceptable
118	11,4	39	13.6	59.8	-	Critical
113	6,3	16	5.6	20.1	-	Acceptable
200	-	-	-	-	72	Critical
180	-	-	-	-	156	Critical
172	-	-	-	-	180	Critical
200	-	-	-	-	163	Critical
168	-	-	-	-	260	Acceptable

4. SUMMARY AND CONCLUSIONS

The relation between the ASTM flow and the deformation from the stability test is not presented here but was not as strong. The coefficient of determination was about 0.70.

The relations between different workability tests on mortar were investigated; a standardize flow test of the ASTM, a small-scale test of the well-known Abrams cone test usually used for concrete and a specially designed stability test aiming to simulate an issue of the 3D printing process. Four different admixtures were tested in a two-level full-factorial design. Linear regressions were computed to observe relationships between the results and draw conclusions.

When all mixes are involved, the slump of Abrams cone is well linearly related to the ASTM flow, but not as much as with the deformation from the stability test. Therefore, conducting the Abrams cone test with mortar could give clues about the way the mixture should behave under the load of the stability test. Consequently, it should help understanding the way the mortar reacts in a 3D printing context supporting the load of several other mortar layers.

This study point out that the fact that the flow of the mortar being a test harder to implement in situ because of its need for specific installation (a flow table), the Abrams cone could give information about the capacity to flow of the mortar. In fact, the real scale Abrams cone is already widely used for testing concrete slump; the utilization of a smaller one for mortar should not be an issue. Moreover several values of characteristics of different mixtures were highlighted in order to narrow the acceptable ones for printing.

Nevertheless, when regression is conducted on one admixture exclusively, results show that for each pair of tests highlighting X mixes, the coefficient of determination drops (Tables 4 and 5). This makes us notice that conventional tests with emerging admixture like the CSH-seed has a less predictable effect on workability. Moreover the way the X is incorporated into the mix has to be changed, a later addition will be tried.

5. RECOMMANDATIONS

The competition effects between the admixtures have not been studied in this paper. As a result, some discrepancies might be elucidated thanks to an adsorption study.

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